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<p>(54) Title: RHEOLOGY MODIFIED COMPOSITIONS AND PROCESSES THEREOF</p> <p>(57) Abstract</p> <p>Rheology modified compositions, and methods for forming the compositions, are disclosed. The compositions and methods are useful in obtaining desirable properties, including viscosity, in cosmetic, pharmaceutical or household product formulations.</p>		

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## RHEOLOGY MODIFIED COMPOSITIONS AND PROCESSES THEREOF

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The present invention is directed to rheology modified compositions, and to methods for forming the compositions. The compositions and methods are useful in applications including cosmetics, pharmaceuticals, and household products.

### 10 BACKGROUND OF THE INVENTION

For many applications, including personal care, pharmaceuticals, and household products, it is desirable that compositions for use in the intended applications have a desired viscosity. In particular, a number of applications require that the compositions used therefor have a relatively high viscosity. The rheology  
15 can affect properties such as, for example, ease of application, ease of handling, esthetic appeal, product stability and retention of a product where applied.

Personal care formulations such as lotions and creams can contain large amounts of solvent. Such formulations often use an oil or emollient as a solvent or a primary component. While oils possess highly desirable properties for cosmetic use,  
20 such as emolliency and the ability to solvate and remove make-up, the presence of oils can be inconvenient in the fluid form and present difficulties in application.

Household products such as cleaning products can contain substantial amounts of oil or wax as solvents or major components. It is generally desirable that a cleaning product applied to a surface, particularly a hard surface, not run over the  
25 surface before it can be wiped. Products such as furniture polish are often oil or wax-based, and should be thick enough to remain where applied so that polishing can be accomplished.

Some of the undesirable properties of oils or emollients can be decreased or eliminated by rheological modification such as thickening of the oil. For example, a  
30 cosmetic formulation can be used in a thickened composition, such as a cream, gel, or a water-in-oil or oil-in-water emulsion, particularly a water-in-oil emulsion.

Preparation of water-in-oil emulsions generally requires thickening of the continuous oily phase of the emulsion. Thickening of the oily phase can also be required in preparing cosmetic gels, particularly anhydrous gels.

The presentation of a formulation in the form of an anhydrous gel is useful, for example, if components of the formulation are sensitive to moisture and/or to oxygen. Certain ingredients in cosmetic or pharmaceutical formulations are unstable in water. For example, vitamin C degrades in water relatively rapidly. Thus, such unstable ingredients are advantageously provided in anhydrous formulations such as an anhydrous gel. Oily compositions, such as oily dispersions, are suitable anhydrous formulations; however, in addition to other difficulties associated with oily compositions as discussed herein, such unstable ingredients can be insoluble or insufficiently soluble in oily formulations.

Polymers are known for use as thickeners or rheology modifiers in personal care, household and pharmaceutical applications. However, certain polymers can be incompatible with other components in the formulations, such as fragrances in cosmetic formulations. Thickening of oils can also be accomplished by incorporation of silicas, bentonites or metal salts of fatty acids such as aluminum salts, or esterified derivatives of sugars such as dextrin palmitate. Thickening has also been accomplished by incorporation of a wax in the oily phase. However, creams thickened by simply adding waxes, silicas or bentonites can have a texture that is unappealing or difficult to use.

U.S. Patent No. 5,318,995 discloses a method for thickening a water-in-oil emulsion by using copolymers containing a slight amount of ionic or ionizable groups.

European Patent No. EP 550,745 discloses thickening of the oil phase of a cosmetic composition using a combination of two copolymers.

The use of polysaccharide alkyl ethers for thickening oil-based compositions is described in European Patent Application No. 795,322. However, the disclosure provides that the process is generally applicable to certain oils having solubility parameters within a specified range.

Thus, a need continues for methods and compositions useful in thickening

solvent-based formulations for personal care, pharmaceutical, and household uses. There is also a need for compositions that have the advantageous properties of oils but with minimization or elimination of undesirable properties. The present invention is directed to these, as well as other, important ends.

5

## SUMMARY OF THE INVENTION

One aspect of the present invention is a composition comprising a rheology modifier, and a solvent mixture comprising a non-polar oil or wax and a miscible hydrogen-bonding compound, wherein the solvent mixture has a polar solubility parameter of less than 6.5 Joules per cubic centimeter (J/cc)<sup>1/2</sup>. In preferred  
10 embodiments, the rheology modifier is a modified polysaccharide. In more preferred embodiments the rheological modifier is a polysaccharide alkyl, alkenyl, alkynyl, aryl, arylalkyl, or arylalkenyl ether, or a mixture thereof.

Another aspect of the present invention is a composition comprising a  
15 rheology modifier, and a solvent mixture comprising a non-polar oil of animal, petroleum or vegetable origin, or a wax, and a miscible hydrogen-bonding compound. In preferred embodiments, the non-polar oil or wax comprises one or more of: a silicone oil, a mineral oil, an aliphatic hydrocarbon, a natural wax, a petroleum wax, or a synthetic derivative of a natural or petroleum wax. In preferred  
20 embodiments, the rheology modifier is a modified polysaccharide. In more preferred embodiments the rheological modifier is a polysaccharide alkyl, alkenyl, alkynyl, aryl, arylalkyl, or arylalkenyl ether, or a mixture thereof.

A further aspect of the present invention is a process for forming a composition. The process includes combining a miscible hydrogen-bonding  
25 compound and a modified polysaccharide at ambient temperature to form a mixture. In preferred embodiments, the rheology modifier is a polysaccharide alkyl, alkenyl, alkynyl, aryl, arylalkyl, or arylalkenyl ether. The mixture is allowed to stand at ambient temperature until solvation occurs. A non-polar oil or wax is then added to the solvated mixture to form a substantially uniform composition.

30 Another aspect of the present invention is a process for forming a silicone oil composition. The process includes combining a miscible hydrogen-bonding

compound and a modified polysaccharide, then adding, at ambient or elevated temperature, a silicone oil to form a substantially uniform composition. In preferred embodiments, the modified polysaccharide ether is a polysaccharide alkyl, alkenyl, alkynyl, aryl, arylalkenyl, or arylalkyl ether.

5 Another aspect of the present invention is an emulsion containing a non-polar oil or wax, a rheology modifier, and a miscible hydrogen-bonding compound, wherein the solvent mixture has a polar solubility parameter of less than  $6.5 \text{ (J/cc)}^{1/2}$ . In preferred embodiments, the rheology modifier is a modified polysaccharide. In more preferred embodiments, the modified polysaccharide is polysaccharide alkyl,  
10 alkenyl, alkynyl, aryl, arylalkyl, or arylalkenyl ether, or a mixture thereof. The emulsion can be an oil-in-water emulsion or a water-in-oil emulsion. In preferred embodiments, the emulsion is an water-in-oil emulsion.

A further aspect of the present invention is a personal care formulation containing a non-polar oil or wax, a rheology modifier, and a miscible hydrogen-  
15 bonding compound, wherein the solvent mixture has a polar solubility parameter of less than  $6.5 \text{ (J/cc)}^{1/2}$ . In preferred embodiments, the rheology modifier is a modified polysaccharide. In more preferred embodiments, the modified polysaccharide is a polysaccharide alkyl, alkenyl, alkynyl, aryl, arylalkyl, or arylalkenyl ether, or a mixture thereof. The personal care formulation can be applied to the hair, skin, mucous  
20 membranes, and/or nails.

Another aspect of the present invention is an anhydrous formulation containing a non-polar oil or wax, a rheology modifier, and a miscible hydrogen-bonding , wherein the solvent mixture has a polar solubility parameter of less than  $6.5 \text{ (J/cc)}^{1/2}$ . In preferred embodiments, the rheology modifier is a modified  
25 polysaccharide. In more preferred embodiments, the rheology modifier is an alkyl, alkenyl, alkynyl, aryl, arylalkyl, or arylalkenyl ether, or a mixture thereof.

## DETAILED DESCRIPTION OF THE INVENTION

The present invention provides improved oil-containing compositions  
30 for personal care, pharmaceutical, or household applications, and methods for making the compositions. It has been surprisingly and unexpectedly found that the

use in such compositions of a rheology modifier and a hydrogen-bonding compound can provide compositions having desired properties, such as viscosity, for use in the intended applications, while reducing undesirable properties often observed in oil-containing compositions. It has further been unexpectedly found that the  
5 compositions and methods of the present invention can be used to provide improved optical clarity, as evidenced by measurable decreases in turbidity, of compositions containing organic solvents and polymers. A further unexpected property of the methods and compositions of the present invention is that they can provide improved properties, such as increased viscosity and improved optical clarity, when  
10 used at room temperature. That is, although heating may be advantageously used for example, to speed mixing of components of the compositions of the present invention, it has been unexpectedly discovered that heating is not necessary to effect uniformity and/or clarity of the compositions. Thus, the compositions of the present invention can be prepared at ambient temperature.

15 The methods and compositions of the present invention can be used, if desired, to provide personal care, pharmaceutical, or household compositions that are solid at room temperature. The use of the compositions and methods of the present invention in formulating solid compositions can provide for reduction of constraints during formulation. For example, it is often necessary that formulations  
20 intended to be solid at room temperature be rapidly solidified in order to ensure uniformity and minimize sedimentation. According to the present invention, such rapid solidification may not be required.

Thus, in one embodiment, the invention provides methods for improving optical clarity in oil-containing compositions. Personal care and other formulations  
25 that include thickening agents often have an undesirable amount of turbidity. It has been surprisingly and unexpectedly found that the use of a hydrogen-bonding compound in combination with a rheology modifier provides compositions having desired properties for personal care, pharmaceutical or household applications, including viscosity, while also exhibiting reduced turbidity as compared to thickened  
30 compositions not containing hydrogen-bonding compounds.

In another embodiment, the invention provides methods for forming, at

ambient temperature, oil-based compositions containing polymeric viscosity-modifying agents. It has been surprisingly and unexpectedly found that the use of a hydrogen-bonding compound, such as an alcohol, reduces or eliminates the need for heat in dissolving the viscosity-modifying agent in the oily phase.

5           The methods and compositions of the invention are useful, for example, in forming anhydrous formulations. Such anhydrous formulations can contain ingredients that are unstable, insoluble, or minimally soluble in water, such as, for example, vitamins including vitamins A, C, D and E.

          "Personal care compositions", as used herein, includes all oil-containing  
10   formulations generally intended for use in the care and/or protection of the skin, hair, nails, mucous membranes, oral cavity or body and generally not used for medicinal or curative effects. As used herein, "personal care" compositions includes cosmetic compositions.

          "Household compositions" and "household formulations", as used herein,  
15   includes all products used by a consumer in cleaning, repairing, maintaining, remodeling, beautifying, decorating, caring for, improving, or sanitizing a residence and its appliances or contents. contents include kitchen and bathroom fixtures, furniture, dishware, cookware, and clothing.

          "Pharmaceutical composition", as used herein, includes compositions  
20   comprising drugs, medicaments, medicinal or curative products, tonics, dietary supplements, vitamins, minerals and the like, whether intended to be taken internally or applied topically.

          "Topical application", as used herein, includes application of a personal care or pharmaceutical composition to the skin or mucous membrane. "Dermatological  
25   formulation" or "dermatological treatment", as used herein, includes all formulations and methods intended for use primarily on the skin, based on their effects thereon, and thus encompasses both cosmetic and pharmaceutical applications.

          "Oil", as used herein, includes oils derived from mineral, vegetable, and animal sources. Thus, the term oil is used broadly and includes petroleum oils, such  
30   as long-chain hydrocarbons, and silicone oils. For example, compounds containing linear or branched, saturated or unsaturated hydrocarbon alkyl chains of at least

about 5 carbon atoms, such as 5, 6, 7, 8 or more carbon atoms, are oils according to the present invention. Thus, such compounds containing hydrocarbon chains of at least 8, 9, 10, 11, or 12 carbon atoms up to a preferred upper limit of 28, 27, 26, 25, 24, 23, or 22 carbon atoms are oils according to the present invention.

5 "Wax", as used herein, means an organic compound, preferably a hydrocarbon compound, that is solid at room temperature. In addition to hydrocarbons, waxes include fatty acid-alcohol esters.

"Alkyl", as used herein, includes all saturated hydrocarbon moieties, whether straight, branched, or cyclic. "Alkenyl" includes all hydrocarbon moieties, whether  
10 straight, branched, or cyclic, containing at least one carbon-carbon double bond. "Alkynyl" includes all hydrocarbon moieties, whether straight, branched or cyclic, containing at least one carbon-carbon triple bond.

"Hydrogen-bonding compound", as used herein, includes all organic compounds containing at least one electronegative atom such as oxygen, nitrogen,  
15 or sulfur and capable of forming non-covalent intermolecular bonds by means of the electronegative atoms. Examples include alcohols, amines, and thiols.

"Polar solubility parameter", as used herein, represents the cohesive energy density of a fluid due to dipole-dipole and hydrogen bonding interactions. Calculation of the solubility parameter is described in the discussion of Example 3  
20 below.

Preferred hydrogen bonding compounds according to the invention are those containing one or more hydroxyl groups. Particularly preferred hydrogen bonding compounds are alcohols. Alcohols having a hydrocarbon chain of at least about 8 carbon atoms are preferred, alcohols having a hydrocarbon chain of from about 8 to  
25 about 32 carbon atoms are more preferred, and alcohols having a hydrocarbon chain of from about 10 to about 24 carbon atoms are even more preferred. In particular, in cosmetic applications, alcohols having from 12 to 24 carbon atoms are highly preferred. Guerbet alcohols of total chain length from about 12 to about 32 carbon atoms are well known in the art for use in personal care applications, and are  
30 useful in the present invention. Guerbet alcohols are 2-alkyl alkanols, such as, for example, 2-butyl octanol, 2-hexyl decanol, 2-octyl dodecanol. Examples of

particularly preferred alcohols for use in the present invention are decanol, dodecanol, hexadecanol, stearyl alcohol, butyl octanol, hexyldecanol, and octyldodecanol. If desired, a mixture of two or more hydrogen bonding compounds, such as a mixture of alcohols, can be used.

5 Hydrogen bonding compounds, such as alcohols, can be present in the compositions of the invention in amounts from about 0.1 to about 60 weight percent, preferably from about 0.2 to about 40 weight percent, more preferably from about 0.3 to about 20 weight percent, even more preferably from about 0.5 to about 10 weight percent, based on the total weight of the oil phase of the composition.

10 Preferably, the hydrogen bonding compounds are present at from about 1 to about 5 weight percent, more preferably from about 1 to about 4 weight percent, and still more preferably from about 1 to about 3 weight percent.

Rheology modifiers, according to the invention, are preferably thickening agents, which increase the viscosity of liquids. Rheology modifiers include synthetic  
15 polymers and polysaccharides. Modified polysaccharide alkyl, alkenyl, alkynyl, aryl, arylalkyl or arylalkenyl ethers are highly preferred. The modified polysaccharide alkyl, alkenyl, alkynyl, aryl, arylalkyl or arylalkenyl ethers can be neutral or charged.

"Modified", as used herein to describe polysaccharides includes polysaccharide alkyl, alkenyl, alkynyl, aryl, arylalkyl or arylalkenyl ethers, esters,  
20 urethanes, amides, carbonates or polysaccharides having any other linkage to the polysaccharide backbone. The modified polysaccharides can have additional substituents which can be cationic, anionic or nonionic. Examples of cationic substituents include quaternary ammonium and phosphonium groups. Examples of anionic substituents include carboxyl, phosphate, sulfate, carboxyalkyl, sulfoalkyl,  
25 and phosphoalkyl. Examples of nonionic substituents include alkyl, aryl, and hydroxyalkyl. Also useful as an additional substituent is a substituent group formed by reaction of the polysaccharide ether with an alkyl ketene dimer or with an alkyl succinic anhydride. The additional substituent or substituents can be the same as or different from the first alkyl, alkenyl, alkynyl, aryl, or arylalkyl group. Of the modified  
30 polysaccharides, neutral polysaccharides are preferred and of these, modified polygalactomannans are highly preferred. Polygalactomannans are known in the art

and are polymers comprising, as polymerized units, galactose and mannose. Polygalactomannans differ from each other, *inter alia*, in the ratio of galactose units to mannose units. The galactose:mannose ratio is determined, in part, by the source of the galactomannan. Polygalactomannans can be obtained from several sources, including guar, tara, fenugreek, and locust bean gum. For the present invention, modified polygalactomannans derived from guar gum, such as alkyl guar, and especially ethyl guar, are particularly useful. Modified polygalactomannans preferably have a degree of substitution of greater than 2.4. However, other modified polygalactomannans can be advantageously used in the compositions and methods of the present invention. Synthetic polymers useful as rheology modifiers include acrylic acid polymers and copolymers, vinylic polymers such as polyvinyl pyrrolidone polymers and copolymers, polyvinyl acetates, and polyalkenes that are oil soluble.

"Miscible", as used herein, means able to dissolve uniformly in a liquid.

"Oily phase", as used herein, refers to a uniform phase, in a uniform or non-uniform composition, which contains the oil or wax. For certain applications, as will be recognized by those skilled in the art, waxes will be preferred. For example, lipstick formulations often contain one or more waxes or solid oils. Similarly, in other applications, oils that are liquids at room temperature will be preferred.

In compositions of the present invention, the oily phase can comprise any oil or mixture of oils conventionally employed in personal care, household and pharmaceutical formulations. Such oils include, but are not limited to: hydrocarbons, including the mineral oils, such as the paraffin oils, the petrolatum oils, hydrogenated polyisobutylene, linear or branched hydrocarbons, and triglycerides, particularly vegetable oils. Examples of hydrocarbon oils for use in the present invention include isooctane, isododecane, and isohexadecane. Examples of vegetable oils include sunflower seed oil, sesame seed oil, linseed oil, tung oil, oiticica oil, soybean oil, cottonseed oil, coconut, rapeseed oil, sweet almond oil, calophyllum oil, palm oil, avocado oil, jojoba oil, olive oil, castor oil, and grain germ oils such as wheat germ oil. Other oils include various oily esters derived from a long-chain acid and/or alcohol or polyol, such as purcellin oil; capric/caprylic triglyceride, isopropyl, butyl or

cetyl myristate; isopropyl, butyl or ethyl-2-hexyl palmitate; isopropyl, butyl octyl, hexadecyl or isocetyl stearate; decyl oleate; hexyl laurate; propylene glycol dicaprylate; diisopropyl adipate; and the mixtures of C<sub>12</sub>-C<sub>15</sub> alkyl benzoate esters commercially available under the trademark FINSOLV™ TN from Finetex. Also  
5 suitable are animal oils, such as perhydrosqualene; and silicone oils, such as dimethylpolysiloxanes, phenyldimethicones, trimethyl phenyl silsesquioxane, decamethylcyclopentasiloxane, dodecamethylcyclohexasiloxane, octamethylcyclotetrasiloxane, dimethylcyclasiloxanes, cyclomethicones, alkyldimethicones, and mixtures thereof. Silicone oils are commercially available, for  
10 example, from Dow Corning Corporation. Preferred oils, according to the invention, are those having more than about 8 carbon atoms and include, but are not limited to, jojoba, sesame, corn, olive, safflower, and rape seed oils; shea butter, and synthetic oils such as purcellin oil, 2-ethyl hexyl palmitate, 2-octyldodecyl stearate, 2-octyldodecyl erucate, isostearyl isostearate, 2-octyldodecyl benzoate, hydrogenated  
15 isoparaffin, and silicone oils.

According to the present invention, the rheology modifier is present in an amount sufficient to achieve the desired thickness for the intended application. In general, it is present in an amount of from about 0.1% to about 40%, preferably from about 0.3% to about 30%, more preferably from about 0.4% to about 20%, even  
20 more preferably from about 0.5 to about 10% by weight, based on the weight of the oil. If the composition is a neat composition, the rheology modifier is present in an amount from about 0.5% to about 8% by weight, based on the weight of the oil. Neat composition, as used herein, is intended to mean a composition that is essentially free of water. If the composition is an emulsion composition, such as, for  
25 example, a water-in-oil emulsion, the rheology modifier is present in an amount from about 0.5 to about 10 % by weight, based on the weight of the oil. As will be recognized by those skilled in the art, the amount of rheology modifier for a particular personal care, household or pharmaceutical application will be determined, in part, upon the form in which the composition is to be provided.

30 The compositions and methods of the present invention are useful in a wide variety of products, including personal care, household, and pharmaceutical

products. The compositions of the invention can be provided in various forms, such as a liquid, a gel, a solid bar, an emulsion, a paste, an aqueous/alcoholic lotion or dispersion, or an ointment, and can optionally be packaged as an aerosol or dispersed as a foam.

5       The compositions of the invention can contain various additives known in the art including additional rheology modifiers, emulsifiers, thickening agents, such as polyacrylic acids, cellulose derivatives or esters of fatty acids and of polyethylene glycol, sequestering agents, foam reinforcers, preservatives, fragrances, electrolytes, fatty substances such as fatty alcohols, ceramides or mineral,  
10 vegetable, animal or synthetic oils or waxes. Amounts of additives and other ingredients can be, for example, from about 0.001% to 20% by weight of the composition. When the compositions of the instant invention are personal care compositions, they may optionally contain additives conventionally used in the personal care industry, including but not limited to active ingredients, perfumes,  
15 preservatives, fragrance retention agents, fragrance releasing gels, and sunscreen agents. Such additives and their use in personal care compositions are well known in the art and can be added by known techniques before, during, or after the thickening of the oil. Other ingredients well known to those skilled in the art can also be incorporated into the personal care compositions. Such ingredients include  
20 coloring agents, opacifiers, UV screening agents, antioxidants, pearlescence agents, biocides, antibacterial agents, antidandruff agents, antiseborrheic agents, antiparasitic agents, repellents, dyes, pigments, oxidizing agents, reducing agents, moisturizers, anionic, nonionic or cationic polymers, vitamins, alpha -hydroxy acids, tocopherol and its esters, fatty esters of ascorbic acid, 18- beta -glycyrrhetic acid,  
25 and ceramides.

When the compositions of the present invention are provided in an emulsion, the emulsion can contain any ingredients known in the art for use in emulsions, such as surfactants and/or dispersing aids. Emulsions can also contain thickeners, which can be, for example, water-soluble or water-swellaable polymers contained primarily  
30 within the aqueous phase of the emulsion. Examples of suitable water-soluble or water-swellaable polymers include: hydroxyethylcellulose,

hydroxypropylmethylcellulose, cetyl modified hydroxyethylcellulose, agar, carrageenans, pectins, guar, hydroxypropylcellulose, crosslinked acrylic acid polymers and copolymers, polyethylene glycols, oxyethylene polymers and copolymers, vinylcaprolactam homopolymers and copolymers, vinylmethyl ether co-maleic anhydride polymers, and polyvinylpyrrolidone.

Powders can be incorporated into compositions of the invention. Examples of powders include chalk, talc, kaolin, starch, smectite clays, chemically modified magnesium aluminum silicate, organically modified montmorillonite clay, hydrated aluminum silicate, fumed silica, aluminum starch octenyl succinate and mixtures thereof.

Personal care products made using the compositions and methods of the present invention are useful in a variety of personal care applications such as, for example, treatment of the skin including moisturizing, cleaning, softening, protecting, hydrating, or smoothing the skin; conditioning, softening, cleansing, or styling the hair; minimizing transepidermal water loss; conditioning or treating the nails, or delivering ingredients including sunscreens, vitamins, pigments, dyes, or tints to the hair, skin and/or nails.

Personal care products for which the compositions and methods of the present invention are suitable include, for example, moisture retention agents, moist towels, towels for cleansing or moisturizing the skin, towels for make-up application or removal, hair styling agents, skin conditioners, hair conditioners, deodorants, including spray, aerosol, gel and stick deodorants, antiperspirants, including spray, aerosol, gel and stick antiperspirants, sunscreen creams or lotions, emollient lotions, milks or creams, make-up removal creams lotions or milks, foundation bases, lotions, milks or creams for artificial tanning, shaving creams, shaving foams, shaving lotions, after shave lotions, face masks, make-up products for the eyes, lipsticks, lip glosses, colors and foundations for the face, shampoos, bath or shower products, hand cleaning products, and compositions for dyeing, bleaching, curling, or straightening the hair. In products such as towels for use in make-up removal or application, the compositions of the present invention can be incorporated within and/or carried on the surface of the towel. Other personal care products, for which

the compositions and methods of the present invention are useful, include dentifrices, denture cleaners, denture adhesives, scalp treatment products, blemish treatment products, wound dressings, wound care and treatment products, and oral care products. Such personal care products are generally commercially available to the consumer without a prescription and can be used to treat minor injuries and conditions. Personal care products can optionally contain active ingredients such as antiseptics, anesthetics, and antibiotics, and can be applied to the skin or to the mucous membranes, and can be contained in mucoadhesive compositions.

The methods and compositions of the present invention can provide advantages in the use and application of personal care formulations. For example, substantially uniform compositions of the invention can provide for improved distribution of ingredients throughout a formulation, thus providing for improved delivery, including more uniform delivery, of effective or active ingredients to the hair, skin, or nails. Thus, vehicles for the delivery of cosmetic ingredients can advantageously contain compositions of the invention. Examples of ingredients for which the compositions and methods of the invention can provide improved delivery include sunscreens, fragrances, pigments, and agents for treatment of the skin such as those described elsewhere herein. Thus, for example, the methods and compositions of the invention can provide enhanced protection of the skin or hair from the sun by providing for improved delivery of sunscreens. This can provide for enhanced protection from the sun, and for protection from ultraviolet and/or infrared radiation, depending in part upon the absorption properties of the sunscreen and/or other radiation-absorbing ingredients in the composition. It is also expected that the methods and compositions can provide for improved delivery of fragrance to the skin, thus enhancing the longevity of fragrance on the skin.

The compositions and methods of the present invention are applicable to household formulations. In such applications, the compositions and methods of the invention can provide advantages in addition to those already mentioned above, in particular, more uniform delivery of effective or active ingredients.

Household products for which the compositions and methods of the invention are useful include cleaning products, pretreatment products, products used in

remodeling or decorating the home, and sterilizing products. Also included are pet care and grooming products. Also included within "household products" and "household formulations" are products used in cleaning, repairing, decorating or remodeling an automobile, boat, bicycle or other vehicle, and products used in  
5 repairs done in a residence by a consumer, such as lubricants for sawing, drilling, or milling wood or metal. Also included are lubricants for household machinery or equipment such as sewing machines and other small appliances. Examples of cleaning products include detergents, stain removers, bleach, glass cleaner, fabric softeners including fabric softener papers, cloths or sponges, oven pretreatment,  
10 oven cleaner, cleaners for hard surfaces such as bathroom fixtures and tiles, furniture cleaners and polishes, and cleaners or polishes for wood floors. Remodeling and decorative products include paints, inks, dyes, wood stains, varnishes, and shellacs.

For household applications, other ingredients, in addition to those already  
15 mentioned herein, can be present. For example, for household applications, the compositions can contain buffering agents, chelating agents, codispersants, surfactants, enzymes, fluorescent whitening agents, electrolytes, builders, antioxidants, thickeners, fragrance, dyes, colorants, pigments, defoamers, or mixtures thereof. Further examples of optional ingredients include bleach  
20 scavengers, sodium perborate, reducing sugars, short chain alcohols; solvents and hydrotropes such as ethanol, isopropanol and xylene sulfonates; flow control agents, enzyme stabilizing agents; soil suspending agents; antiredeposition agents; anti-tarnish agents; anti-corrosion agents; colorants; abrasives, and other functional additives. The pH of the cleaning composition may be adjusted by addition of strong  
25 acid or base. Such buffering agents include sodium carbonate and sodium borate.

The compositions and methods of the present invention are also useful in pharmaceutical applications. Pharmaceutical applications generally involve the delivery of a drug or other pharmacologically active agent. As used herein, the term "drug" or "pharmacologically active agent" or any other similar term means any  
30 chemical or biological material or compound that induces a desired biological or pharmacological effect, including (1) affecting living processes, (2) having a

prophylactic effect on an organism and preventing an undesired biological effect such as preventing an infection, (3) treating a disease, including alleviating a condition caused by a disease, for example, alleviating pain or inflammation caused as a result of disease, and/or alleviating, reducing, or completely eliminating the disease from the organism. Pharmaceutical applications thus include materials useful in treating diseases or other conditions of the skin, such as, for example, acne, rashes, fungal, viral or bacterial infections, cracking, peeling, burns, lesions, and dermatitis. Pharmaceutically active agents that can be delivered using formulations comprising the compositions of the present invention, or made according to the methods of the present invention, include antiseptics, anesthetics, vitamins and/or minerals especially those prescribed for medical reasons, and drugs for the treatment of disease. Treatment of disease can be accomplished by administering to a patient in need of such treatment a pharmaceutical composition comprising a composition of the present invention. Such administration can include oral administration, topical application, or administration by means of a suppository.

In pharmaceutical applications, the compositions can include such pharmaceutically acceptable additives, ingredients, carriers, extenders, fillers, processing aids, and excipients known in the art. The term "pharmaceutically acceptable carriers" is meant to include any solvents, dispersion media, coatings, and adsorption-delaying agents. Thus, the compositions of the invention can contain conventional excipients known for use in the preparation of pharmaceutical formulations, such as thickening agents, emulsifiers, flavoring agents, coloring agents, and the like.

Examples of pharmaceutical applications for which the compositions and methods of the present invention are useful include formulations intended for ingestion and formulations intended for topical application. Ingestible pharmaceutical formulations for which the present invention is useful include dosage forms such as capsules, caplets, tablets, liquids, and gels. Topically applied pharmaceutical formulations for which the invention is useful include creams, lotions, gels, adhesives, ointments, and formulations for transdermal delivery of pharmaceutically active materials. For transdermal delivery, chemical enhancers for

facilitating transport of the drug through the skin or mucosa and other additives can be included in the composition.

Various modifications of the invention, in addition to those described herein, will be apparent to those skilled in the art from the foregoing description. Such  
5 modifications are intended to fall within the scope of the appended claims.

### **EXAMPLES**

The following examples are merely illustrative of the present invention and should not be considered limiting of the scope of the invention in any way. These  
10 examples and equivalents thereof will become more apparent to those skilled in the art in light of the present disclosure and the accompanying claims.

#### **Example 1**

Optical clarity of mixtures containing an alkylgalactomannan, N-Hance®  
15 AG-200 polymer from Hercules Incorporated was determined by turbidity measurements. The mixtures were N-Hance® AG-200/alcohol/organic solvent mixtures in which various levels of polymer (AG-200), alcohol (dodecanol or hexadecanol), and organic solvent (Finsolv TN, isopropyl palmitate, or 50:50 FinsolvTN:isopropyl palmitate) were combined. Mixtures were prepared by first  
20 mixing appropriate weights of the solvent components (note: hexadecanol and cetyl alcohol are solid at room temperature) and adding an appropriate weight of N-Hance® AG-200 alkyl galactomannan. The mixtures were heated to 70-85°C for approximately one hour to speed dissolution of the polymer. Sample turbidity was determined using an Orbeco-Hellige model 966 turbidimeter. Samples were  
25 contained in Wheaton disposable glass scintillation vials. Turbidity data is presented in Table 1.

50:50 Finsolv TN:Isopropylpalmitate										
T (NTU) Wt % Polymer					T (NTU) Wt % Polymer					
%	0.5	1	2	3		%	0.5	1	2	3
dodecanol						hexadecanol				
0.0	10.2	11.9	24.2	50.8		0.0	10.2	11.9	24.2	50.8
1.0	10.1	11.5	23.5	35.6		0.5	9.7	12.3	27.1	35.9
2.0	9.5	11.8	23.4	35.3		1.0	9.7	12.6	27.0	35.9
5.0	5.8	10.8	22.7	33.2		1.5	9.5	11.8	26.6	35.6
10.0	5.5	10.2	21.2	32.1		2.0	8.9	12.1	26.7	35.4
Isopropylpalmitate										
%	0.5	1	2	3		%	0.5	1	2	3
dodecanol						hexadecanol				
0.0	8.5	18.4	38.0	58.1		0.0	8.5	18.4	38.0	58.1
1.0	8.2	17.2	37.2	55.3		0.5	10.0	19.5	43.3	57.5
2.0	8.2	17.4	36.2	54.4		1.0	10.0	18.9	37.9	56.1
5.0	7.4	15.8	33.5	50.8		1.5	9.7	19.0	35.4	55.3
10.0	6.7	13.8	29.2	46.5		2.0	9.5	18.5	37.0	54.4
Finsolv TN										
%	0.5	1	2	3		%	0.5	1	2	3
dodecanol						hexadecanol				
0.0	4.6	10.2	22.5	34.5		0.0	4.6	10.2	22.5	34.5
1.0	4.7	10.0	21.9	34.5		0.5	4.6	10.8	22.0	32.9
2.0	4.5	9.9	21.2	33.7		1.0	4.9	10.7	22.0	33.6
5.0	4.4	9.0	19.6	31.5		1.5	5.2	11.2	22.0	33.2
10.0	4.0	8.1	17.7	28.4		2.0	4.5	10.9	21.5	33.0

It is apparent from the data in Table 1 that, as the concentration of polymer increases, the turbidity of the resulting solution increases. It is also clear the addition of a long chain alcohol can serve to lower the turbidity of the resulting solution, yielding an aesthetically improved result.

5

### Example 2

Mixtures of N-Hance® AG-200 polymer, or N-Hance® AG-50 polymer, and decanol were prepared and allowed to stand. Upon standing, the polymer became highly swollen by the alcohol. At low levels of polymer, the solute completely swelled while at higher levels excess unsolvated polymer remained. Results are presented in Table 2.

10

Table 2

## N-Hance®AG Polymer/Decanol Swelling and Uptake

Wt. % polymer	Wt. % Decanol	Appearance: N-Hance®AG-50 system	Appearance: N-Hance® AG-200 system
10	90	clear gel/viscous polymer lower phase (40), fluid solvent upper layer (60)	clear gel/viscous polymer lower phase (60), fluid solvent upper layer (40)
20	80	clear gel/viscous polymer lower phase (70), fluid solvent upper layer (30)	clear gel/viscous polymer phase, more dilute in polymer towards top
30	70	clear gel/viscous polymer phase, more dilute in polymer towards top	clear gel/viscous polymer phase
40	60	clear gel/viscous polymer phase	clear gel/viscous polymer phase, vary small amount of excess unsolvated polymer
50	50	clear gel/viscous polymer phase, small amount of excess unsolvated polymer	clear gel/viscous polymer phase, appreciable amount of excess unsolvated polymer
60	40	clear gel/viscous polymer phase, appreciable amount of excess unsolvated polymer	clear gel/viscous polymer phase, large amount of excess unsolvated polymer
70	30	clear gel/viscous polymer phase, large amount of excess unsolvated polymer	clear gel/viscous polymer phase, large amount of excess unsolvated polymer
80	20	clear gel/viscous polymer phase, large amount of excess unsolvated polymer	clear gel/viscous polymer phase, large amount of excess unsolvated polymer
90	10	clear gel/viscous polymer phase, large amount of excess unsolvated polymer	clear gel/viscous polymer phase, large amount of excess unsolvated polymer

The results indicate that gels or viscous solutions containing about 30 to 40 weight percent polymer can be prepared.

### Example 3

5

#### Viscosity modification of solvents with N-Hance® AG polymers in the presence of alcohol

A solution was prepared of N-Hance AG® polymer in 2-butyl octanol (25 wt % N-Hance AG® 50 polymer or 20 wt % N-Hance AG® 200 polymer). 1 or 2 grams of  
10 the polymer solution was accurately weight in a scintillation vial. 0 or 2 grams of 2-butyl octanol was accurately weighed into each vial.

Primary solvent was accurately weighed into each vial to yield a final total volume of approximately 20 ml. Primary solvents used were: DC-345 (Dow Corning cyclodimethicone), DC-556 (Dow Corning Phenyl trimethicone), P-99A (Permethyl  
15 Specialties isododecane), P-101A (Permethyl Specialties isohexadecane). The solvent, polymer, and alcohol were mixed with a magnetic stirrer and heated on a hot plate (95°C) for 1 hour to yield nominal polymer concentrations of 1-1.5 and 2-2.5 weight percent with 2-butyl octanol concentrations of 2-10 and 12-24 weight percent.

20 Solution viscosities were measured using a Brookfield LVDV-II+ viscometer with a small sample adapter and a #18 spindle. 8 ml of each sample was added to the sample reservoir of the viscometer and thermostatted at 25°C. The spindle rotation rate was set to the maximum rotation rate that resulted in an on-scale reading.

The sample turbidity was directly measured using an Orbeco-Hellige  
25 turbidimeter.

Solubility parameters were calculated using volume fraction mixing rules ( $\delta_{d,mix} = \phi_{2\text{-butyl octanol}} \delta_{d,2\text{-butyl octanol}} + \phi_{\text{primary solvent}} \delta_{d,\text{primary solvent}}$ ;  $\delta_{p,mix} = \phi_{2\text{-butyl octanol}} \delta_{p,2\text{-butyl octanol}} + \phi_{\text{primary solvent}} \delta_{p,\text{primary solvent}}$ ). Component solubility parameters used are listed in Table 4. Also listed in Table 3 are the turbidity, viscosity, and appearance of each composition.  
30 Solubility parameters used are those described in C.M. Hansen, "The Three-dimensional Solubility Parameters", *J. Paint Technol.*, Vol. 39, p. 105 (1967).

Primary Solvent	Polymer*	Wt. % primary solvent	Wt. % polymer	Wt. % 2-butyl octanol	$\delta_d$ (J/cc) <sup>1/2</sup>	$\delta_{polar}$ (J/cc) <sup>1/2</sup>	Turbidity (NTU)	Viscosity (cP)	Comments
DC-345							2.0	5.4	
DC-556							0.7	25.4	
I-12							0.4	29.7	
P-99A							0.3	4.3	
P-101A							0.4	6.0	
DC-345	AG-50	95.1	1.2	3.7	15.2	0.4	5.9	6.8	Significant amount undissolved solid present
DC-345	AG-50	84.7	1.3	14.0	15.3	1.6	44.2	22.6	Homogeneous solution
DC-345	AG-50	90.3	2.4	7.2	15.3	0.8	180.0	82.2	Homogeneous, hazy solution
DC-345	AG-50	81.6	2.3	16.2	15.4	1.8	65.7	68.9	Homogeneous solution
DC-556	AG-50	94.7	1.3	3.9	17.1	0.5	7.2	71.3	Homogeneous solution
DC-556	AG-50	84.4	1.3	14.3	17.0	1.6	7.4	72.3	Homogeneous solution
DC-556	AG-50	90.0	2.5	7.5	17.1	0.9	15.8	223.2	Homogeneous solution
DC-556	AG-50	80.2	2.5	17.3	17.0	2.0	15.6	237.6	Homogeneous solution
P-99A	AG-50	92.6	1.8	5.5	15.9	0.5	37.8	10.4	Minor amount undissolved solid present
P-99A	AG-50	81.8	1.6	16.7	15.9	1.5	11.6	12.2	Homogeneous solution
P-99A	AG-50	87.7	3.1	9.2	15.9	0.8	31.6	24.9	Homogeneous solution
P-99A	AG-50	76.4	3.0	20.6	15.9	1.9	21.5	26.5	Homogeneous solution
P-101A	AG-50	93.6	1.6	4.8	16.6	0.5	16.6	15.9	Homogeneous solution
P-101A	AG-50	80.8	1.6	17.6	16.5	1.7	11.0	19.5	Homogeneous solution
P-101A	AG-50	87.4	3.1	9.4	16.6	0.9	28.4	55.0	Homogeneous solution
P-101A	AG-50	75.4	2.9	21.7	16.5	2.1	18.8	52.6	Homogeneous solution
DC-345	AG-200	94.1	1.2	4.7	15.2	0.5	245.0	31.6	Appreciable amount undissolved solid present
DC-345	AG-200	84.9	1.0	14.1	15.3	1.5	39.4	32.3	Minor amount undissolved solid present
DC-345	AG-200	89.1	2.2	8.7	15.3	0.9	150.4	152.7	Homogeneous, hazy solution
DC-345	AG-200	80.0	2.0	18.0	15.4	2.0	60.5	116.7	Homogeneous solution
DC-556	AG-200	94.5	1.1	4.4	17.1	0.5	9.1	135.0	Homogeneous solution
DC-556	AG-200	85.5	1.0	13.5	17.0	1.5	8.3	102.0	Homogeneous solution

Primary Solvent	Polymer*	Wt. % primary solvent	Wt. % polymer	Wt. % 2-butyl octanol	$\delta_d$ (J/cc) <sup>1/2</sup>	$\delta_{polar}$ (J/cc) <sup>1/2</sup>	Turbidity (NTU)	Viscosity (cP)	Comments
DC-556	AG-200	90.0	2.0	8.0	17.1	0.9	14.8	514.0	Homogeneous solution
DC-556	AG-200	81.1	1.9	17.0	17.0	1.9	13.8	366.0	Homogeneous solution
P-99A	AG-200	93.1	1.4	5.6	15.9	0.5	21.3	10.0	Homogeneous solution
P-99A	AG-200	80.6	1.3	18.1	15.9	1.6	10.4	13.5	Homogeneous solution
P-99A	AG-200	86.1	2.8	11.1	15.9	1.0	28.8	52.2	Homogeneous solution
P-99A	AG-200	73.4	2.7	23.9	15.9	2.2	21.9	69.5	Homogeneous solution
P-101A	AG-200	93.0	1.4	5.6	16.6	0.5	16.8	40.5	Homogeneous solution
P-101A	AG-200	78.6	1.5	19.8	16.5	1.9	12.0	41.2	Homogeneous solution
P-101A	AG-200	88.1	2.4	9.5	16.6	0.9	25.3	84.9	Homogeneous solution
P-101A	AG-200	76.6	2.3	21.1	16.5	2.0	17.7	98.1	Homogeneous solution

\* "AG-200" = N-Hance® AG 200 alkyl galactomannan polymer; "AG-50" = N-Hance® AG 50 alkyl galactomannan polymer.

5

Table 4

## Solubility Parameters for Solvents Used in Example 3

Solvent	$\delta_{total}$ (J/cc) <sup>1/2</sup>	$\delta_d$ (J/cc) <sup>1/2</sup>	$\delta_p$ (J/cc) <sup>1/2</sup>	Density (g/cc)
Dow Corning DC-345	15.2	15.2	0	0.96
Dow Corning DC-556	17.2	17.2	0	0.98
Isododecane (Permethyl 99A)	15.9	15.9	0	0.747
Isohexadecane (Permethyl P-101A)	16.6	16.6	0	0.79
2-butyl octanol (Jarcol I-12)	17.0	16.12	9.78	0.833
2-hexyl decanol (Isofol 16)	17.0	16.26	8.55	0.836

A  $\delta_p$  value of 0 was assigned to DC-345 and DC-556 based on the solvatochromic response of dimethylsiloxane polymers and low molecular weight linear dimethyl siloxanes relative to linear and cyclic alkanes (see J. E. Brady, D. Bjorkman, C. D.

Herter, P. W. Carr; Analytical Chemistry (1984) 56, 278-83 and references cited therein.).  $\delta_d$  values were calculated from the measured refractive index of the liquids using the relation  $\delta_d = 62.78 \cdot (n^2 - 1)/(n^2 + 2) \text{ (J/cc)}^{1/2}$ , where  $n$  is the refractive index of the pure liquid. The solubility parameters of 2-butyl octanol and 2-hexyl decanol were obtained from European patent No. 0,795,322,A1.

$\delta_{\text{total}} = (\delta_d + \delta_p)^{1/2} = (-\Delta E_{\text{vap}}/V)^{1/2}$  where  $\delta_{\text{total}}$ ,  $\delta_d$ , and  $\delta_p$  are the total, dispersive, and polar solubility parameters, -

#### Example 4

#### 10 Preparation of Gel Antiperspirant

<u>Water-In-Oil Antiperspirant Soft Gel</u>			
	<u>Part</u>	<u>Ingredients</u>	<u>Weight %</u>
	A	REACH? 301 Solution (a)	35.0
15	B	Deionized water	21.0
	C	Cyclomethicone and Dimethicone copolyol (b)	20.0
	D	Glycerin, 99.7%	20.0
	E	Natrosol 250MR CS	0.25
	F	N-Hance® AG	1.00
20	G	2 Hexyl decanol	3.00

#### Procedure:

- Added E to B. Raised pH to 8.5. Mixed for 30 minutes. Added D. Mixed for 30 minutes.
- Added A to the premix prepared in Step 1. Mixed for 15 minutes.
- Very slowly added the premix from Step 2 to C at slow speed.
- Mixed for 15 minutes.
- Mixed F and G for 30 minutes. Very slowly added to the premix from Step 4 at slow speed.

(a) Reheis, Inc.

(b) Silsoft MSC (OSI Specialties)

**Example 5****Preparation of roll-on antiperspirant****I. Control**

5	<b><u>Ingredients</u></b>		
	<b><u>% By Wt. 3X g</u></b>		
	A. Aluminum Zirconium Tetrachlorohydrate GLY Powder (Rezal 36 GP SUF, Reheis)	20.0	60.0
10	B. Cyclomethicone and Quaternium-18 Herculite and SDA 4 (Bentone Gel VS-5/PC, RHEOX)	13.5	40.5
	C. Cyclomethicone (Dow Corning 244)	66.0	198.0
15	D. Silica (Sylox 15, W. R. Grace)	0.5	1.5
	E. Fragrance	<u>qs</u>	<u>0.0</u>
20		100.0g	300.0

**Procedure for Control****Run #1 X31651-67-1A**

- 25
1. Mixed "B" with "C", "E", and "F" for twenty minutes.
  2. Added "A" and mixed for fifteen minutes.
  3. Added "D" and mixed for ten minutes.
  4. Homogenized for three minutes in 4 oz. jar.

## II. Roll-on antiperspirant made according to the invention

ANTIPERSPIRANT SUSPENSION ROLL-ON WITH N-HANCE® AG polymer

<u>Ingredients (% By Wt., 3X g)</u>			
5	A. Aluminum Zirconium Tetrachlorohydrate GLY Powder (Rezal 36 GP SUF, Reheis)	16.0	48.0
	B. Cyclomethicone and Quaternium-18 Herculite and SDA 4 (Bentone Gel VS-5/PC, RHEOX)	13.0	39.0
10	C. Cyclomethicone (Dow Corning 244)	66.0	198.0,
	D. Silica (Sylox 15, W. R. Grace)	0.5	1.5
15	E. Fragrance	qs	0.0
	F. N-Hance® AG	1.0	3.0
	2 Hexyl decanol	<u>3.0</u>	<u>9.0</u>
20		100.0g	300.0g

Procedure

1. Mixed "B" and "F" with "C" for twenty minutes.
- 25 2. Added "A" and mixed for fifteen minutes.
3. Added "D" and mixed for ten minutes.
4. Homogenized for three minutes in 4 oz. jar.

30

**Example 6****Preparation of liquid soap****Opaque Liquid Soap Formula**

<u>Ingredients</u>	<u>Weight %</u>
Water	75.73
Sodium C14-C16 olefin sulfonate, 40% active	7.50
Sodium lauroyl sarcosinate, 30% active	6.66
Cocamidopropyl betaine, 35% active	6.66
Glycol stearate	1.00
Natrosol® Plus 330	0.80
Propylene glycol	0.50
Glycerin	0.50
Tetrasodium EDTA	0.30
Stearalkonium chloride	0.10
Methylparaben	<u>0.25</u>
	100.00

5

**Procedure:**

1. The NP 330 was dispersed in water. The pH was raised to about 8.0 - 8.5 to dissolve the polymer and mixed for 45 minutes. The methylparaben was added to the finished solution.
- 10 2. While slowly stirring the water-soluble polymer solution, the stearalkonium chloride, olefin sulfonate, and glycol stearate were added. The mixture was heated to 80°C until all of the glycol stearate was melted and the solution had turned opaque.
3. The remaining ingredients were added while cooling the solution slowly to room
- 15 temperature.
4. The color and fragrance were added.

**Example 8****Preparation of soap formulation****Toilet Soap Formula**

5	<b><u>Ingredients</u></b>	<b><u>Weight %</u></b>
	Water	65.70
	Sodium C14-C16 olefin sulfonate	20.00
	Sodium lauroyl sarcosinate	10.00
	Cocamide MEA	3.00
	Natrosol® 250HR	1.00
	Disodium EDTA	0.20
	Methylparaben	<u>0.10</u>
		100.00

**Procedure:**

1. The Natrosol 250HR product was dispersed in water. The pH was raised to about 8.0 - 8.5 to dissolve the polymer and mixed for 45 minutes. The methylparaben  
10 was added to the finished solution.
2. In a separate vessel, the surfactants were combined, heated to 80°C, and mix until homogeneous.
3. The surfactant solution was added to the water-soluble polymer solution and mixed until well blended.
- 15 4. The disodium EDTA was added and cooled to room temperature.

### Source and Description of Products Used in Liquid Soap and Toilet Soap Formula

**Generic or**

	<u><b>CTFA Adopted Name</b></u>	<u><b>Trademark</b></u>	<u><b>Supplier</b></u>
5	Stearalkonium chloride Northfield, Illinois	Ammonyx 4002	Stepan Chemical Co.
10	Sodium C14-C16 olefin sulfonate Northfield, Illinois	Bio-Terge AS-40	Stepan Chemical Co.
	Sodium lauroyl sarcosinate	Hamposyl L-30	W. R. Grace & Company Nashua, New Hampshire
15	Cocamidopropyl betaine	Lexaine C	Inolex Chemical Company Philadelphia, Pennsylvania
20	Cocamide MEA Paterson, New Jersey	Monamid CMA	Mona Industries Inc.
	Tetrasodium EDTA Northfield, Illinois	Perma Kleer 100	Stepan Chemical Co.
25	Hydroxyethylcellulose	Natrosol 250HR	Hercules Incorporated
		N-Hance® AG	Hercules Incorporated

### Example 9

### Preparation of Conditioner formulation

### Baby Hair Conditioner Formula

	<u>Ingredients</u>	<u>Weight %</u>
35	Natrosol® 250 HR	1.0
	Water	74.1
	Cetrimonium chloride (25%)	12.2
	Lauramine oxide (30%)	10.2
	Polyquaternium-17 (62%)	1.5
	Propylene glycol	1.0
	Perfume, preservative	q.s. to 100.0

**Procedure:**

1. The Natrosol was dispersed in water with good agitation. The pH was raised to about 8.0 - 8.5 to dissolve the polymer and mixed for 45 minutes or until fully dissolved.
- 5 2. The remaining ingredients were added in the order listed, mixing well between additions.

**Example 10****Preparation of Cream Rinse Formulation**

10

**Pearlescent Cream Rinse Formulation**

	<u>Ingredients</u>	<u>Weight %</u>
	Phase A	
	Natrosol® Plus 330	1.00
15	Natrosol 250HHR	0.30
	Water	82.30
	Phase B	
	Stearalkonium chloride (25%)	10.10
	Propylene glycol	1.50
20	Phenyl trimethicone	1.45
	Alkyl galactomannan	0.01
	2 butyl octanol	0.04
	Oleth-20	1.50
	Polyquaternium-17 (62%)	1.80
25	Perfume, preservative	q.s. to 100.00

**Procedure:**

1. The NP 330 and Natrosol 250HHR were dispersed in water with good agitation. The pH was raised to 8.0 - 8.5; the dispersion was mixed until fully dissolved.
- 30 2. In a separate vessel, the stearalkonium chloride and propylene glycol were mixed together and heated to 80°C.

3. The other ingredients listed in Phase B were added in the order listed to the mixture of stearalkonium chloride and propylene glycol and mixed well between each addition.
4. The surfactant mixture was added to the HMHEC1 solution, mixed well, and
- 5 cooled to 35°C.
5. The perfume and preservative were then added to form the final formulation.

### Raw Materials and Their Sources for

#### Baby Hair Conditioner and Pearlescent Cream Rinse Formula

10	<u>CTFA Adopted Name</u>	<u>Trademark</u>	<u>Supplier</u>
	Quaternium-48	Adogen 470	Sherex Chemical Co., Inc. Dublin, Ohio
15	Oleth-20	Emulphor ON-870	Rhone-Poulenc Cranbury, New Jersey
	Hydrolyzed animal protein	Lexein X-250	Inolex Chemical Company Philadelphia, PA
20	Polyquaternium-17	Mirapol AD-1	Rhone-Poulenc Cranbury, New Jersey
	Cocamidopropylamine oxide	Ammonyx CDO	Stepan Company Northfield, Illinois
25	Lauramine oxide	Ammonyx LO	Stepan Company Northfield, Illinois
30	Cetrimonium chloride	Varisoft E228	Sherex Chemical Co., Inc. Dublin, Ohio
	Stearalkonium chloride	Varisoft SDC	Sherex Chemical Co., Inc. Dublin, Ohio
35	Modified HEC	Natrosol® Plus 330	Hercules Incorporated Wilmington, Delaware
	Hydroxyethylcellulose	Natrosol 250HHR	Hercules Incorporated

**Example 11****Preparation of Hand and Body Lotion**

	<u>Ingredient</u>	<u>Weight %</u>
5	A Natrosol® Plus 330	0.50
	Distilled water	78.25
	Glycerin, USP	2.00
	B Glycol stearate (Emerest 2400)	2.75
10	N-Hance® AG	0.50
	Stearic acid (Industrene 5016K)	2.50
	Mineral oil (Drakeol 7)	2.00
	Acetylated lanolin (Lipolan 98)	0.50
	Cetyl alcohol (Crodacol C95)	0.25
15	C Distilled water	9.50
	Triethanolamine	0.50
20	D Propylene glycol and diazolidinyl urea and Methylparaben and propylparaben (Germaben II)	0.75 100.00

**Procedure:**

1. NP 330 CS was dispersed into well agitated water from Part A. Glycerin was  
25 added with continued mixing and heated to 80°C. Mixed 15 minutes at 80°C.
2. In a separate vessel, ingredients of Part B were blended. The mixture was  
heated to 80°C and mixed well.
3. Part A was added slowly to Part B under good agitation. The temperature of the  
emulsion was maintained to ~80°C with constant stirring.
- 30 4. Ingredients in C were combined and added to the emulsion. Mixed continuously  
while cooling to 40°C.
5. Added Part D (preservative) to emulsion and mixed well.
6. Cooled the emulsion and filled the containers.

**Materials and Suppliers for Hand and Body Lotion**

	<b><u>CTFA Adopted Name</u></b>	<b><u>Trade Name</u></b>	<b><u>Supplier</u></b>
		HMHEC4	Hercules Incorporated Wilmington, DE
5	Glycol Stearate	Emerest 2400	Henkel Corporation Hoboken, NJ
10	Stearic Acid	Industrine 5016K	Witco Corporation Newark, NJ
	Mineral oil	Drakeol 7	Penreco. Karns City, NJ
15	Acetylated Lanolin	Lipolan 98	Lipo Chemicals Patterson, NJ
	Cetyl alcohol	Crodacol C95	Croda Inc. Parsippany, NJ
20	Hydroxyethylcellulose	Natrosol 250 MR	Hercules Incorporated
	Modified HEC		Natrosol® Plus 330 Hercules Incorporated
25			

		Shaving Cream	
		<u>Ingredients</u>	<u>Amount, g</u>
		Deionized water	790.0
		Sodium hydroxide (24.6% solution)	9.6
5		Potassium hydroxide (34.2% solution)	34.2
		Stearic acid, double pressed	71.6
		Coconut acid	10.0
		Propylene glycol	27.0
		Lauramide DEA	10.0
10		Coconut oil	2.0
		N-Hance® AG	0.5
		Tallow glycerides	30.0
		Preservative (Germaben II)	5.0
		Natrosol® 250 HR	<u>10.0</u>
15		<b>Total</b>	<b>1000.0</b>

**Procedure:**

To prepare the shaving cream concentrate, the sodium hydroxide and potassium hydroxide were added to the deionized water in a vessel at room temperature. The temperature of the vessel was then raised to 75°C and stirred for 5 minutes. The stearic acid and coconut acid were separately pre-melted and then each was added to the caustic/water mixture and then stirred for 30 minutes followed by cooling to 55°C. Natrosol 250 HR was slurried in the propylene glycol and added to the mix. Next, one at a time, lauramide DEA (melted), coconut oil, tallow glycerides (melted), N-Hance AG, and a preservative were added to the vessel and stirred for 15 minutes and allowed to cool.

The concentrate was transferred to container. For the aerosol cream, weigh 225 g into a standard 12-oz shaving cream can. The can is then sealed with a valve assembly using laboratory canning equipment and charged with 9.0 g of propellant.

### List of Ingredients and Their Suppliers for Shave Cream

	<u>CTFA Adopted Name</u>	<u>Trademark</u>	<u>Supplier</u>
5		HMHEC3	Hercules Incorporated Wilmington, Delaware
	Stearic acid	Industrene 5016	Witco Corporation Memphis, Tennessee
10	Coconut acid	Industrene 328	Witco Corporation Memphis, Tennessee
15	Lauramide DEA	Standamid LD	Henkel Corporation Ambler, Pennsylvania
	Coconut oil	Coconut oil	Sigma Chemical Co. St. Louis, Missouri
20	Tallow glycerides	Peacock Acidless Tallow	Geo. Pfau s Sons Co. Jeffersonville, Indiana
	Sorbitol	Sorbo (70% active)	ICI Americas, Inc. Wilmington, Delaware
25	Propylene glycol (and) Diazolidinyl urea (and) Methylparaben (and) Propylparaben	Germaben II	Sutton Laboratories Chatham, New Jersey
30	88/12 Isobutane/propane	A-46 Propellant	Aeropres Corporation Shreveport, Louisiana
35	Propylene glycol	Propylene Glycol	Eastman Chemical Co. Rochester, New York
	Hydroxyethylcellulose	Natrosol® 250HR	Hercules Incorporated
40		N-Hance® AG	Hercules Incorporated

## Standard Cream Toothpaste with HMHEC2

	<u>Ingredient</u>	<u>Wt. %</u>
5	I. Natrosol® 250 HNF	0.75 <sup>1</sup>
	Glycerin 100%	12.50
	N-Hance® AG	0.50
	Sorbitol (70% solids)	16.86
	Distilled water	14.71 <sup>2</sup>
10	II. Dicalcium phosphate, anhydrous	45.00
	III. Tetra sodium pyrophosphate (TSPP)	0.42
15	Sodium saccharin	0.20
	Sodium monofluorophosphate (SMFP)	0.76
	Sodium benzoate	0.50
	Distilled water	6.25
20	IV. Flavor	0.55
	Sodium lauryl sulfate	<u>1.00</u>
		100.00

**Procedure:**

1. The salts of Part III were added to the water in a vessel while stirring and heated to about 60°C to dissolve. The vessel was covered during heating to prevent moisture loss.
- 25 2. Part I. The glycerine was weighed into a beaker and the polymer Natrosol N-Hance AG was dispersed in the glycerin while stirring for about 5 minutes or until adequately dispersed. Sorbitol was added and the mixture was continuously stirred for another 10 minutes. Water was added and stirred for an additional 15 to 30 minutes, making sure that the polymer was completely hydrated (no gels).
- 30 A warm salt solution was added while stirring continuously for an additional 15 minutes or until homogenous (no lumps or gels). This mixture was then transferred to a toothpaste mixer (Ross double planetary mixer).
- 35 3. Part II. The DCP and water were added to a mixer and mixed for 10 min. at a low speed to completely wet the DCP. The mixer was then opened and the paste mixture was scraped from the beaters and bowl sides. The mixer was then

closed and a vacuum was applied. The mixer was run on high speed under vacuum for 20 minutes or until the paste mixture had a smooth consistency.

4. Part IV. The SLS was added to the mixer and mixed for 5 minutes at low speed without vacuum. The flavor was added to the mixer and mixed for 2 min. at low speed. The mixer was then opened and the beaters and bowl sides were scraped down. The mixer was closed and a vacuum was applied and mixed at medium speed for 15 minutes, observing for foaming.
5. The mixer was then shut off and the vacuum was broken and the formulation was packed out as a paste.

<sup>1</sup> Correct polymer weight for moisture content.

<sup>2</sup> For water: Adjust the amount of water for moisture in the polymer.

		Gel	
	<u>Ingredient</u>	<u>Weight %</u>	<u>Weight (g)</u>
A	N-Hance® AG	2.00	8.00
B	Phenyl trimethicone	90.00	360.00
C	2 butyl octanol	<u>8.00</u>	<u>32.00</u>
		100.00	400.00

#### Procedure:

1. The N-Hance AG was added to the blend of "A", "B", and "C".
2. Next, the temperature was raised to 90°C and mixed for one hour.
3. Cooled to room temperature while mixing.
4. The preservative methylparaben was added and mixed for 10 minutes.
5. Dispensed into containers.

#### Gel

Dow Corning 345 was used in place of phenyl trimethicone.

### Denture Adhesive

One hundred-gram batches of denture adhesive were prepared according to the following formulas:

5	Mineral Oil	20.0
	Petrolatum	30.0
	HMHEC2	47.0
	<u>N-Hance® AG</u>	<u>3.0</u>
10		100.0

#### Procedure:

The petrolatum and mineral oil were weighed into a 250 ml beaker. The beaker was placed in a circulating oil bath heated to 67°C. The contents were stirred at speed 0 with an electric mixer having two 1½" dia. propellers spaced ¼" apart on the shaft. When contents were 65°C, the polymer was added slowly while adjusting the mixer speed to maintain a vortex in the mixture. Mixing was continued for one hour.

### Clear Stick Antiperspirant

A two phase method was used to prepare the clear stick antiperspirant as follows:

#### Phase I

About 65% of the total propylene glycol used (excluding that which is part of the antiperspirant salt solution) was charged to a reaction vessel. Klucel® GF was added to the vessel and stirred well until dissolved. The vessel was heated to dissolve the polymer. Once the polymer was dissolved, the solution was heated to 110°C-115°C, and the dibenzylidene sorbitol was added and mixed until completely dissolved. This Phase I solution was then cooled to about 100°C.

30

**Phase II**

About 35% of the total propylene glycol used (excluding that which is part of the antiperspirant salt solution) was added to the another vessel, stirred and heated to about 60-70°C. The Na<sub>4</sub>EDTA was added and mixed well to form a slurry. The antiperspirant salt solution was added next to this vessel and the solution was mixed well until it becomes clear and homogeneous. The emollients, dimethicone copolymer, was added and the Phase II solution was mixed until it became clear.

**Combined Phase:**

Phase II was added to Phase I while mixing and cooled to 80°C. Optionally, a fragrance would be added at this point and allowed to mix well. The product was poured into a 1 oz. glass jars and allowed to cool overnight. After cooling overnight, the samples were tested for physical and chemical properties.

**Equipment Used:**

Two 400 ml glass beakers, oil bath, clamps, mechanical stirrer, Jiffy stirrer and thermometer, and a covering to prevent contamination, such as plastic wrap.

**Total Formulation for this Example:**

20	1. Propylene glycol	46.67g
	2. Al/Zr tetrachlorohydrate-gly	36.60g*
	3. Dibenzylidene sorbitol	0.50 g
	4. Klucel GF	0.30g
	5. Sodium EDTA	0.20g
25	6. Phenyl trimethicone	3.00g
	7. N-Hance® AG	0.05g
	8. 2 hexyl decanol	<u>0.20g</u>
		87.52

\* 30% active solution.

**Phase I:**

	Polypropylene glycol	32.07 g
	Dibenzylidene sorbitol	0.50 g
	Klucel® GF	<u>0.30 g</u>
5		32.87g

**Phase II:**

	Polypropylene glycol	14.60 g
	Al/Zr tetrachlorohydrate-gly	36.60 g
10	Sodium EDTA	0.20 g
	Phenyl trimethicone	3.00 g
	N-Hance® AG	0.05 g
	2 hexyl decanol	<u>0.20 g</u>
		54.65g

15

**Clear Stick Antiperspirant**

DC-345 (cyclodimethicone) was used in place of phenyl trimethicone (DC-556).

**Raw Materials and Their Sources for Antiperspirant Stick**

20	<b><u>Raw Material</u></b>	<b><u>Supplier</u></b>
	Propylene glycol (USP Grade)	EM Science Gibbstown, NJ
25	Al/Zr tetrachlorohydrate-gly Westchlor A2Z 8160 30% PG solution	Westwood Chemical Corporation Middletown, New York
	Dibenzylidene sorbitol Millithix 925	Milliken Chemicals
30	Klucel® GF	Hercules Incorporated Wilmington, DE
	Cyclodimethicone DC-345	Dow Corning
35	Sodium EDTA Aldrich #5403EJ	Aldrich Chemical Company Milwaukee, Wisconsin
	Phenyl trimethicone DC-556	Dow Corning

**Pearlescent Shampoo**

	<u>Ingredient</u>	<u>Weight %</u>	<u>Weight (g)</u>
	Distilled water	q.s. to 100.00	347.25
	TEA-lauryl sulfate (40% active) (Stepanol WAT)	15.00	75.00
5	Sodium lauroamphoacetate (and) sodium trideceth sulfate (Miranol MHT)	10.00	50.00
	Cocamide DEA (Ninol 40C0)	2.50	12.50
	Glycol stearate (Emerest 2400)	1.20	6.00
	Propylene glycol (and) diazolidinyl urea (and)		
10	methylparaben (and) propylparaben (Germaben II)	0.75	3.75
	Natrosol® 250 HHR	0.60	3.00
	N-Hance® AG	0.50	2.50
	Citric acid (50% solution)	<u>pH adjust</u>	<u>-----</u>
		100.00	500.00

15

**Procedure:**

1. The N-Hance® was dispersed by adding slowly to the vortex of well-agitated water in the container. The pH was reduced to 7.0 with citric acid solution to promote dissolution of the surface-treated N-Hance. Heated to 50°C.
- 20 2. Natrosol was slowly sifted into the N-Hance solution and mixed until fully dissolved.
3. The temperature was raised to 70°C. Next, the TEALS and glycol stearate were added, one at a time. Between each addition the mixture was well agitated. Heat was turned off once it looked homogeneous. The mixing was continued.
- 25 4. When the temperature reached 55°C, the remaining ingredients were added, one at a time.
5. Adjusted to pH 5.0 with citric acid solution.
6. Cooled to 40°C and added fragrance.

## List of Ingredients and Their Suppliers for Pearlescent Shampoo

	<u>CTFA Adopted Name</u>	<u>Trade Name</u>	<u>Supplier</u>
5	TEA-lauryl sulfate	Stepanol WAT	Stepan Company Northfield, Illinois
	Sodium lauroamphoacetate (and) sodium trideceth sulfate	Miranol MHT	Rhone-Poulenc Cranbury, NJ
10	Cocamide DEA Northfield, IL	Ninol 40C0	Stepan Company
	Glycol stearate	Emerest 2400	Henkel Corporation Hoboken, NJ
15	Propylene glycol (and) diazolidinyl urea (and) methylparaben (and) propylparaben	Germaben II	Sutton Lab Chatham, NJ
20	Alkyl galactomannan	N-Hance® AG	Hercules Incorporated Wilmington, DE
	Hydroxyethylcellulose	Natrosol® 250 HHR	Hercules Incorporated

<u>Sunscreen Lotion</u>			
	<u>Ingredient</u>	<u>Weight %</u>	<u>Weight (g)</u>
	A. 2 hexyl decanol	0.50	2.50
5	Mineral Oil (Klearol, Witco)	13.00	65.00
	Polyoxypropylene 15 Stearyl Ether (Arlamol E, ICI)	6.00	30.00
	Octyl Methoxycinnamate (Neo Heliopan AV, H&R)	5.00	25.00
	Benzophenon-3 (Uvinul M40, BASF)	3.00	15.00
	Hydrogenated Castor Oil (Castor Wax, Ross)	1.40	7.00
10	Sorbiton Monoisostearate (Arlacel 987, ICI)	1.20	6.00
	Polyoxyethylene Polyol Fatty Acid Ester (Arlatone T, ICI)	1.00	5.00
	Ozokerite Wax (O Wax 77W, Ross)	1.00	5.00
	Polyoxyethylene Fatty Acid Ester (Arlacel 989, ICI)	0.50	2.50
15	B N-Hance® AG	0.50	2.50
	Distilled Water	63.60	318.00
	Glycerine	2.50	12.50
	Magnesium Sulfate	0.70	3.50
	Diazolidinyl Urea, PG, Methylparaben, Propylparaben	0.10	0.50
20	(Germaben II, ISP)	100.00	500.00

**Procedure:**

1. Mixed all ingredients in Part A.
2. Raised temperature to 70°C, stirred for 30 minutes.
- 25 3. For Part B, N-Hance AG was dispersed in water. The slurry pH was raised to 8.5 with NaOH. Mixed until dissolved. Glycerin, magnesium sulfate and the preservative were added one at a time while mixing. The mixture was mixed between each addition to make sure there are no lumps.
4. Part B was added to Part A slowly while stirring.
- 30 5. Stirred 30 minutes at 70°C.
6. Cooled to room temperature while stirring.
7. Filled the containers

### Sunscreen Lotion

Cyclodimethicone (DC-345) was used in place of 50% of mineral oil.

#### 5                      Materials and Their Suppliers for Sunscreen Lotion

	<u>CTFA Adopted Name</u>	<u>Trade Name</u>	<u>Supplier</u>
10	Mineral Oil	Klearol	Witco Corporation Dublin, OH
	Polyoxypropylene 15 Stearyl Ether	Arlamol E	ICI Surfactants Wilmington, DE
15	Octyl Methoxycinnamate	Neo Heliopan AV	H&R Corporation Springfield, NJ
	Benzophenon-3	Uvinul M40	BASF Corporation Washington, NJ
20	Hydrogenated Castor Oil	Castor Wax	Ross
	Sorbiton Monoisostearate	Arlacel 987	ICI Surfactants
25	Polyoxyethylene Polyol Fatty Acid Ester	Arlatone T	ICI Surfactants
	Ozokerite Wax	O Wax 77W	Ross
30	Polyoxyethylene Fatty Acid Ester	Arlacel 989	ICI Surfactants
	N-Hance® AG		Hercules Incorporated Wilmington, DE
35	Diazolidinyl Urea, PG, Methylparaben, Propylparaben	Germaben II	Sutton Labs Chatham, NJ

## Hydro-Alcoholic Roll-On

	<u>Part</u>	<u>Ingredients</u>	<u>Weight %</u>	<u>Weight (g)</u>
	A	REACH 501 Solution		
5		(50% Al chlorohydrate)	39.00	156.00
	B	Procetyl AWS (PPG-5 ceteth-20)	2.00	8.00
	C	HMHEC4	0.20	0.80
	D	Deionized water	15.70	62.80
	E	SD Alcohol 40	41.10	164.40
10	F	N-Hance® AG	1.00	4.00
	G	Fragrance (d)	<u>1.00</u>	<u>4.00</u>
			100.00	400.00

**Procedure:**

- 15 1. HMHEC4 was dispersed into D. Raised pH to 8.5 with NaOH solution. Mixed 30 minutes.
2. Gradually A was added. Mixed rapidly using overhead stirring to dissolve.
3. In a separate container B, E, and F were combined, then added slowly with constant agitation to the rest of the batch.
- 20 4. Added fragrance. Mixed 5 minutes.
5. Pour into roll-on containers.

## Materials and Their Suppliers for Hydro-alcoholic Roll-on

	<u>CTFA Adopted Name</u>	<u>Trade Name</u>	<u>Supplier</u>
5	Aluminum chlorohydrate	REACH 501	Rehies Incorporation Berkeley Height, NJ
	PPG-5 ceteth-20	Procetyl AWS	Croda Incorporation Parsippany, NJ
10		HMHEC4	Hercules Incorporated Wilmington, DE
	Ethyl alcohol	SD Alcohol 40	
15	Fragrance Classic oriental/spice	#Q-7148 Fragrances Inc.	Quest International Mount Olive, NJ
	Hydroxyethylcellulose	Natrosol® 250MR CS	Hercules Incorporated Wilmington, DE
20	Alkylated galactomannan	N-Hance® AG	Hercules Incorporated

## Shower Gel

	<u>Ingredient</u>	<u>Weight %</u>	<u>Weight (g)</u>
	A Deionized water	q.s. to 100.00	276.60
	B Natrosol® 250 HR	0.95	4.75
5	C Sodium Laureth Sulfate (Steol CS460, Stepan)	11.53	57.65
	Disodium Laureth Sulfosuccinate (Stepan Mild SL3, Stepan)	11.80	59.00
	Disodium Cocoamphodiacetate (Miranol C2M Conc NP, Rhone-Poulenc)	6.00	30.00
10	Sodium Lauroyl Sarcosinate (Crodasinc LS 30, Croda)	7.25	36.25
	Propylene Glycol	2.00	10.00
	Quaternized Wheat Protein (WheataFlor, Croda)	1.00	5.00
	Hydrolysate and Hydrolyzed Wheat Protein and Wheat Germ Oil & Polysorbate 20		
15	Glycol Distearate and Laureth-4 and CAPB (Euperlan PK 3000, Henkel)	2.00	10.00
	Disodium EDTA (EDETA BD, BASF)	0.10	0.50
	Perfume (Drom 229033, Drom)	0.35	1.75
	Phenoxyethanol and Methylparaben and Ethylparaben and Propylparaben and Butylparaben (Phinonip, Nipa)	0.60	3.00
20	Phenyl trimethicone (DC-556)	1.00	5.00
	N-Hance® AG	0.02	0.10
	2 hexyl decanol	<u>0.08</u>	<u>0.40</u>
		100.00	500.00

25

**Procedure:**

1. Natrosol was dispersed into well agitated water.
2. The pH of the slurry was raised to 8.5 with an NaOH solution. Mixed until the solution had no lumps. Next, the ingredients of Phase C were added in the order listed above. Mixed for one minute between each addition or until became homogeneous.

30

3. Adjusted pH of the final product to 5.3-5.7.

4. Filled the containers.

### Raw Materials and Their Sources For Shower Gel

	<u>CTFA Adopted Name</u>	<u>Trade Name</u>	<u>Supplier</u>
5	Guar Hydroxypropyltrimonium Chloride	N-Hance® 3196	Hercules Incorporated Wilmington, DE
10	Sodium Laureth Sulfate	Steol CS460	Stepan Company Northfield, NJ
	Disodium Laureth Sulfosuccinate	Stepan Mild SL3	Stepan Company
15	Disodium Cocoamphodiacetate	Miranol C2M Conc NP	Rhone-Poulenc Cranbury, NJ
	Sodium Lauroyl Sarcosinate	Crodasinic LS 30	Croda Incorporated Parsippany, NJ
20	Quaternized Wheat Protein	WheataFlor	Croda Incorporated
25	Hydrolysate and Hydrolyzed Wheat Protein and Wheat Germ Oil & Polysorbate 20 Glycol Distearate and Laureth-4 and CAPB	Euperlan PK 3000	Henkel Corporation Hoboken, NJ
	Disodium EDTA	EDETA BD	BASF Corporation Washington, NJ
30	Perfume	Drom 229033	Drom International Towaco, NJ
35	Phenoxyethanol and Methylparaben and Propylparaben and Butylparaben	Ethylparaben and Phinonip	Nipa Hardwick Inc. Wilmington, DE
	Guar Hydroxypropyl trimoniumchloride	N-Hance 3196	Hercules Incorporated Wilmington, DE
40	Phenyl trimethicone	DC-556	Dow Corning

Cream Make-Up with Sunscreen			
	<u>Ingredients</u>	<u>Trade Name/Supplier</u>	<u>Weight %</u>
5	Phase A	Cyclomethicone (and)	6.0
		Dow Corning® 3225C	
		Dimethicone Copolyol	Formulation Aid
		Decyl Oleate	2.5
		Ceraphyl 140/Van Dyk	
10		Octyl Dimethyl PABA	2.0
		Glyceryl Stearate S.E.	1.0
		Cerasynt Q/Van Dyk	
		Stearic Acid, Triple-Pressed	2.0
		Myristyl Myristate	1.0
15		Mink Wax	1.3
		Alkyl Galactomannan	0.2
		N-Hance AG-200/Hercules	
	Phase B Water		57.3
20	15	Preservative	0.2
		Dowcil 200/Dow Chemical Co.	
		PEG-8	5.0
		Glycereth-26	5.0
		Liponic EG-1/Lipo	
25		Tetrahydroxypropyl	0.5
		Quadrol/BASF	
		- Ethylenediamine	
25	Phase C	Pigment Grind (13% iron	
		Oxide, 40% TiO <sub>2</sub> ,	
		47% Kaolin)	10.0
25		Bentonite	3.0
		Volclay, Superfine	
		Super Pearl 100	3.0
		Mearl	
			100.0

**Procedure:**

Micronized the pigment grind and added the remaining Phase C ingredients.

- 30 Combined Phase C and Phase B. Added Phase B and C mixture to Phase A at 70°C (158°F) while mixing with a cage mixer or side-sweep mixer. Stir-cooled at 28°C (82°F)

## Liquid Make-Up

	<u>Ingredients</u>	<u>Trade Name/Supplier</u>	<u>Weight %</u>
5	Phase A		
	Cyclomethicone	Dow Corning® 344	10.0
	Isodecyl Oleate	Ceraphyl 140A/Van Dyk	2.7
	Stearoxytrimethylsilane	Dow Corning 580 Wax	2.5
	and Stearyl Alcohol		
	Stearic Acid		2.0
10	Glyceryl-Monostearate S.E.	Cerasynt Q/Van Dyk	1.0
	Soya Stearol	Generol 122/Henkel	0.4
	Alkyl Galactomannan	N-Hance AG-200/Hercules	0.2
	Phase B		
	Water		56.0
15	PEG-8	Polyglycol E-400	5.0
	Glycereth-26	Liponic EG-1/Lipo	5.0
	Tetrahydroxypropyl	Quadrol/BASF	0.5
	Ethylenediamine		
	Preservative		0.2
20	Pigment Grind (13% iron		
	Oxide, 40% TiO <sub>2</sub> ,		
	47% Kaolin)		<u>14.5</u>
			100.0

25 **Procedure:**

Micronized the pigment grind and combined with Phase B. Heated both Phase A and Phase B separately to 75°C (167°F) while mixing with a cage mixer or side-sweep mixer. Cooled with mixing. At 48°C (118°F) product begins to become thicker and should be packaged at about 38°C (100°F).

30

**Make-Up Cover Stick**

	<u>Ingredients</u>	<u>Trade Name/Supplier</u>	<u>Weight %</u>
5	Phase A		
	Stearamide MEA	Monamid S/Mona	18.6
	Stearamide MEA Stearate	Witcamide MAS/Witco	8.3
	2-Octyldodecanol		2.0
10	Phase B		
	Alkyl Galactomannan	N-Hance® AG-200/Hercules	
	8.5		
	Mineral Oil		25.0
	Petrolatum		4.0
15	PPG-3 Myristyl Ether	Witconol APM/Witco	8.0
	Pigment Grind		25.0
15	Phase C		
	Preservative		0.1
	Fragrance		<u>q.s.</u>
			100.0

**Procedure:**

- 20 Combined Phase B ingredients at 50°C (122°F) with propeller agitation, then heated to 80°C (176°F). Heated Phase A to 80°C and added to Phase B, mixing until uniform. Reduced temperature to 60°C (140°F) and added Phase C. Casted into stick molds and cooled to room temperature.

## Hair Dressing

			<u>Weight %</u>		
<u>Ingredients Trade Name/Supplier</u>			<u>A</u>	<u>B</u>	<u>C</u>
5	Alkyl Galactomannan	N-Hance AG-200/Hercules	0.5		
	Phenyl Dimethicone	Dow Corning® 556 Fluid	7.5	5.0	
	Dimethicone Copolyol	Dow Corning® 193	5.0		
	Surfactant				
	Petrolatum	Sonojell #9/Witco	31.0	25.0	31.0
10	Mineral Oil	Klearol/Witco	23.0	25.0	
	Mineral Oil and	Nimelesterol D/	47.0		
	Lanolin Oil	Emery Ind.			
	Paraffin Wax			12.0	
	Microcrystalline Wax	Amber Wax/Bareco	12.0		13.0
15	Lanolin		25.0	25.0	
	Mink Wax	Mink Wax/Emulan Inc.	4.0		4.0
	Isopropyl Myristate	Emerest 2314/Emery Ind.		5.0	
	Fragrance		<u>q.s.</u>	<u>q.s.</u>	<u>q.s.</u>
			100	100	100

20

**Procedure:**

**Formulation A** - heated petrolatum and microcrystalline wax until melted. Added remaining ingredients.

**Formulation B** - heated petrolatum and paraffin wax until melted. Added remaining ingredients.

25

**Formulation C** - melted together microcrystalline wax, petrolatum and mink wax. In a separate container, mixed Nimelesterol and Dow Corning 556 fluid. Added to first mixture.

**Eye Shadow Stick Base**

<b><u>Ingredients</u></b>	<b><u>Trade Name/Supplier</u></b>	<b><u>Weight %</u></b>
5 Alkyl Galactomannan	N-Hance® AG-200/Hercules	0.5
Cyclomethicone	Dow Corning® 345 Fluid	29.5
Lanolin Oil	Fluilan/Croda	6.7
Carnauba	Carnauba Wax/Strahl & Pitsch	8.0
Beeswax	Beeswax, White/Strahl & Pitsch	3.3
10 Mineral Oil	Carnation/Witco	22.7
Cetyl Alcohol	Adol 52/Sherex Chemical Co.	2.7
Pigments		<u>26.6</u>
		100.0

**Procedure:**

- 15 Mixed together all waxes and oils and heated until all the waxes were melted. Milled pigments into the base. Poured into molds.

**Creamy Lipstick**

	<u>Ingredients</u>	<u>Trade Name/Supplier</u>	<u>Weight %</u>
	Pigment Grind		
	Titanium Dioxide		4.0
5	Pigment		3.4
	Castor Oil		25.0
	Wax Base		
	Trimethylsiloxysilicate (and)	Dow Corning® 580 Wax	4.5
	Stearyl Alcohol		
10	Myristyl Myristate		7.0
	Octyldodecanol		2.0
	Lanolin Oil		7.0
	Alkyl Galactomannan	N-Hance® AG-200/Hercules	1.0
	Candelilla Wax		5.5
15	Beeswax USP		4.5
	Carnauba Wax		2.5
	Ozokerite (76°C [169°F])		3.5
	Avocado Oil		29.6
	Propylene Glycol (and)	Tenox 20/Eastman	0.1
20	t-Butyl Hydroquinone (and)		
	Citric Acid		
	Preservative	Propyl Paraben	0.1
	DC Red 21		0.15
	Fragrance		<u>1.0</u>
25			100.85

**Procedure:**

- Dissolved the DC Red 21 and fragrance in the silicone wax in advance. Weighed in other ingredients and heated to 80°C (176°F) to melt waxes. Added silicone wax/fragrance blend at 80°C (176°F) and poured into molds. Molds should be
- 30 chilled prior to removal of sticks to avoid crushing or bruising of sticks.

**Hair Relaxers**

			<u>Weight %</u>	
	<u>Ingredients</u>	<u>Trade Name/Supplier</u>	<u>A</u>	<u>B</u>
5	Phase A Trimethylsilyl-	Dow Corning® Q2-8220	2.0	
	Amodimethicone	Conditioning Additive or		
		Dow Corning® X2-8230		2.0
	Alkyl Galactomannan	N-Hance® AG-200/Hercules	0.5	0.5
	Emulsifying Wax	Polawax/Croda	7.0	7.0
10	Cetyl Alcohol	Adol 52/Sherex Chemical Co.	1.0	1.0
	Petrolatum	Protopet White IS/Witco	4.0	4.0
		Sonneborn		
	Mineral Oil			
	Carnation/Witco Sonneborn		15.0	15.0
15	Phase B Water		64.5	64.5
	Propylene Glycol		2.0	2.0
	Phase C Sodium Hydroxide (50%)		<u>4.0</u>	<u>4.0</u>
20			100.0	100.0

**Procedure:**

Separately, heated Phase A and Phase B to 72°C (161°F). Added Phase B to Phase A slowly with agitation. Added Phase C. Homogenized.

25

**Fragrance Sachet**

	<u>Ingredients</u>	<u>Trade Name/Supplier</u>	<u>Weight %</u>
Phase A	Cyclomethicone (and) Dimethicone Copolyol	Dow Corning® 3225C Formulation Aid	45.0
5	Stearyl Alcohol		3.5
	Glyceryl Tribehenate	Syncrowax HR-C/Croda	11.0
	Alkyl Galactomannan	N-Hance® AG-200/Hercules	1.0
	Isofol-16 (2-hexyldecanol)		3.0
10	Phase B	Water	17.5
	Phase C	Fragrance	<u>10.0</u>
			100.0

**15 Procedure:**

Mixed and heated the oil and water phases separately to about 75°C (167°F).

Slowly added the water to the oil phase with good agitation. Stirred-cooled to 55°C (131°F) and added fragrance. Required hot fill. Must be packaged in tightly sealed container. Unperfumed product melts at approximately 55°C (131°F). Perfume

20 additions will lower this melting point.

**Water-in-Silicone Antiperspirant Pump Spray**

	<u>Ingredients</u>	<u>Trade Name/Supplier</u>	<u>Weight %</u>
Phase A	Isofol-16		2.0
25	Cyclomethicone	Dow Corning® 345 Fluid	33.0
	Mineral Oil		2.0
	Phenyl Trimethicone	Dow Corning® 556 Fluid	5.0
	Cyclomethicone (and) Dimethicone Copolyol	Dow Corning® 3225C Formulation Aid	7.9
30	Alkyl Galactomannan	N-Hance® AG-200/Hercules	0.1
Phase B	Polysorbate 20	Tween 20/ICI	1.0
	Aluminum Chlorohydrate (and) Water		<u>49.0</u>
35			100.0

**Procedure:**

Mixed the Phase A ingredients in a suitable vessel and measured the phases refractive index. In a separate vessel, mixed the Phase B ingredients and measured the refractive index. Adjusted the refractive index of Phase A appropriately. When the refractive indices matched, mixed Phase A with a Gifford-Wood Eppenbach mixer using moderate shear and slowly added to Phase B. When all of Phase B is added, sheared the emulsion to uniformity. It is suggested that high one-pass shearing be applied to improve stability.

10

**Clear Water-in-Silicone Antiperspirant Roll-On**

	<u>Ingredients</u>	<u>Trade Name/Supplier</u>	<u>Weight %</u>
	Phase A		
	Cyclomethicone	Dow Corning® 344 Fluid	20.0
15	Cyclomethicone (and)	Dow Corning® 3225 C	8.0
	Dimethicone Copolyol	Formulation Aid	
	Alkyl Galactomannan	N-Hance® AG-200/Hercules	0.5
	Isofol-16		1.5
20	Phase B		
	Aluminum Zirconium		58.0
	Tetrachlorohydrate-Gly		
	(and) Water		
	Water		3.3
	Propylene Glycol		<u>8.7</u>
25			100.0

**Procedure:**

Mixed the Phase A ingredients in a suitable vessel and measured the phases refractive index. In a separate vessel, mixed the Phase B ingredients and measured this phases refractive index. Adjusted the refractive index of Phase B to match Phase A by adding propylene glycol to increase its refractive or adding water to

lower it. When the refractive indices matched, mixed Phase A with a Gifford-Wood Eppenbach mixer using moderate shear and slowly added to Phase B. When all of Phase B is added, sheared the emulsion to uniformity.

**What is claimed is:**

1. A composition comprising a rheology modifier, and a solvent mixture comprising a non-polar oil or wax and a miscible hydrogen-bonding compound, wherein the solvent mixture has a polar solubility parameter of less than  $6.5 \text{ (J/cc)}^{1/2}$ .  
5
2. The composition of claim 1 wherein the non-polar oil or wax is selected from the group consisting of silicone oils, mineral oils, aliphatic hydrocarbons, natural waxes, petroleum waxes, and synthetic derivatives of natural or petroleum waxes.  
10
3. The composition of claim 1 wherein the rheology modifier ether is a modified polysaccharide.
4. The composition of claim 3, wherein the modified polysaccharide is a polysaccharide comprising a moiety selected from the group consisting of alkyl, alkenyl, alkynyl, aryl, arylalkyl and arylalkenyl.  
15
5. The composition of claim 4 wherein said moiety is linked to the polysaccharide backbone by a linkage selected from the group consisting of ethers, esters, urethanes, amides, and carbonates.  
20
6. The composition of claim 3 wherein the modified polysaccharide is a modified galactomannan.
7. The composition of claim 6 wherein the modified galactomannan is an ethyl guar.  
25
8. The composition of claim 1 wherein the polar solubility parameter of the solvent mixture is less than about 4.  
30
9. The composition of claim 1 wherein the polar solubility parameter of

the solvent mixture is less than about 2.

10. The composition of claim 1 wherein the polar solubility parameter of the solvent mixture is less than 1.

5

11. The composition of claim 1 wherein the polar solubility parameter of the solvent mixture is less than 1.

12. An emulsion comprising the composition of claim 1.

10

13. A composition for the treatment of the skin, comprising the composition of claim 1.

14. A composition according to claim 13, further comprising at least one member selected from the group consisting of sunscreens, vitamins, pigments, moisture retention agents, skin conditioners, fragrance retention agents, and fragrance releasing gels.

15

15. A method for treating the skin, comprising applying to the skin a cosmetic formulation comprising the composition of claim 1.

20

16. A method for providing enhanced protection from the sun, comprising applying to the skin a composition comprising the composition of claim 1.

25

17. A method for minimizing transepidermal water loss comprising applying to the skin a composition comprising the composition of claim 1.

18. A cosmetic composition comprising the composition of claim 1, wherein said hydrogen-bonding compound is an alcohol.

30

19. The composition of claim 18 wherein the alcohol is 2-alkyl alkanol.

20     The composition of claim 1, wherein said hydrogen bonding compound  
is an amine.

21.     The composition of claim 1, wherein said hydrogen bonding compound  
5     is a thiol.

22.     An oral care composition comprising the composition of claim 1.

23.     A dentifrice comprising the composition of claim 1.

10

24.     A denture adhesive comprising the composition of claim 1.

25.     A towel for make-up application or removal, comprising the  
composition of claim 1.

15

26.     A towel for cleansing or moisturizing the skin, comprising the  
composition of claim 1.

27.     A method for treating disease, comprising administering to a patient in  
20     need of treatment a pharmaceutical composition comprising the composition of claim  
1.

28.     The method of claim 27 wherein the disease is a disease of the skin.

25     29.     The method of claim 28 wherein the composition is applied topically.

30.     A wound dressing comprising the composition of claim 1.

31.     An antiperspirant or deodorant composition comprising the  
30     composition of claim 1.

32. The composition of claim 31 wherein the antiperspirant or deodorant is in the form of a clear or opaque stick.

33. The composition of claim 31 wherein the antiperspirant or deodorant is in the form of a pump spray, aerosol, or roll-on.

34. An anhydrous composition comprising at least one vitamin and the composition of claim 1.

35. A mucoadhesive composition comprising the composition of claim 1.

36. A lipstick or lip gloss comprising the composition of claim 1.

37. A liquid makeup composition comprising the composition of claim 1.

38. A sunscreen composition comprising the composition of claim 1.

39. A hair care composition comprising the composition of claim 1.

40. A skin care composition comprising the composition of claim 1.

41. An ointment comprising an anesthetic, antiseptic, or antibiotic comprising the composition of claim 1.

42. A pharmaceutical composition comprising a drug and a pharmaceutically acceptable carrier comprising the composition of claim 1.

43. The composition of claim 42, in the form of a tablet, or capsule.

44. A method for enhancing the longevity of fragrance on the skin,

comprising applying to the skin a composition comprising a fragrance and the composition of claim 1.

45. A process for forming a composition, comprising combining a miscible  
5 hydrogen-bonding compound and a rheology modifier at ambient temperature to form a mixture;

allowing the mixture to stand at ambient temperature until solvation occurs;  
and

adding a nonpolar oil or wax to the solvated mixture to form a substantially  
10 uniform composition.

46. The process of claim 44 wherein the non-polar oil or wax is selected  
from the group consisting of silicone oils, mineral oils, aliphatic hydrocarbons,  
natural waxes, petroleum waxes, and synthetic derivatives of natural or petroleum  
15 waxes.

47. The process of claim 44 wherein the rheology modifier ether is a  
modified polysaccharide.

20 48. The process of claim 46, wherein the modified polysaccharide is a  
polysaccharide comprising a moiety selected from the group consisting of alkyl,  
alkenyl, alkynyl, aryl, arylalkyl and arylalkenyl.

49. The process of claim 47 wherein said moiety is linked to the  
25 polysaccharide backbone by a linkage selected from the group consisting of ethers,  
esters, urethanes, amides, and carbonates.

50. The process of claim 47 wherein the modified polysaccharide is a  
modified galactomannan.

30

51. The process of claim 49 wherein the modified galactomannan is an

ethyl guar.

52. A process for forming a composition, comprising:  
combining a miscible hydrogen-bonding compound and a polysaccharide  
5 alkyl, alkenyl, alkynyl, aryl, arylalkyl or arylalkenyl ether; and adding, at ambient  
temperature, a silicone oil to form a substantially uniform composition.

53. The process of claim 51 wherein the non-polar oil or wax is selected  
from the group consisting of silicone oils, mineral oils, aliphatic hydrocarbons,  
10 natural waxes, petroleum waxes, and synthetic derivatives of natural or petroleum  
waxes.

54. The process of claim 51 wherein the rheology modifier ether is a  
modified polysaccharide.

15

55. The process of claim 53, wherein the modified polysaccharide is a  
polysaccharide comprising a moiety selected from the group consisting of alkyl,  
alkenyl, alkynyl, aryl, arylalkyl and arylalkenyl.

- 20 56. The process of claim 54 wherein said moiety is linked to the  
polysaccharide backbone by a linkage selected from the group consisting of ethers,  
esters, urethanes, amides, and carbonates.

57. The process of claim 53 wherein the modified polysaccharide is a  
25 modified galactomannan.

58. The process of claim 56 wherein the modified galactomannan is an  
ethyl guar.

- 30 59. An ink composition comprising the composition of claim 1.

# INTERNATIONAL SEARCH REPORT

Int. l. Application No

PCT/US 99/21210

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 A61K7/00

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 0 804 924 A (OREAL) 5 November 1997 (1997-11-05) claims 1-13	
A	EP 0 795 323 A (OREAL) 17 September 1997 (1997-09-17) claims 1-17	
A	EP 0 795 322 A (OREAL) 17 September 1997 (1997-09-17) cited in the application claims 1-22	
A	EP 0 281 360 A (HI TEK POLYMERS INC) 7 September 1988 (1988-09-07) abstract	

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

### \* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

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Date of the actual completion of the international search

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Information on patent family members

International Application No

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